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Maximize Column Lifetime Using Supelco Carrier Gas Purifiers

Abstract

Oxygen, moisture, and hydrocarbon contaminants in carrier gas will lower GC performance and shorten column life. Even brief exposure to these carrier gas contaminants can damage small sections of the column. Once damage occurs, column degradation is irreversible. To insure maximum column life, you must protect it from carrier gas impurities at all times. Sources of carrier gas contamination include the gas cylinder, the cylinder changing process, fittings and regulators. Use of Supelco's recommended

purifier products will eliminate carrier gas contaminants and maximize column life.

Sources of Carrier Gas Contamination

There are many sources of oxygen, moisture, and hydrocarbon contaminants. The primary source is the carrier gas cylinder. All grades of gas contain contaminants. The differences are in the level and types of contaminants measured. There is always a chance that even the highest purity gas contains contaminants that can damage your column. Al-

though use of high-purity gases reduces the risk of rapid damage, impurities in all grades of cylinder gas will shorten the life of your column.

A second source of contaminants is the cylinder changing process. Contaminants enter regulators and gas lines while they are disconnected. No matter what the cylinder changing process, a small amount of these contaminants unavoidably reaches the column when the system goes back on line. This results in the damage of a small section of the column. Once damaged, the column degradation process is irreversible.

A third source of contamination is from regulators, valves, and fittings in the system. Contaminants permeate through all regulator diaphragms unless they are made of metal. Valves and fittings present potential sources of leaks from connection and re-connection. Dirt and occasional over compression prevents proper reseal of the fitting. If fittings and tubing are made of different materials, leaks de-



velop over time because of differing rates of expansion. Leaking fittings are an ongoing source of contaminants that shortens column life.

The Solution is Gas Purifiers

As long as contaminants are present in the GC system, columns will not last as long or perform as well as they should. To guard against this, the system must contain components designed to eliminate damaging contaminants. Gas purifiers are the only sure way to guarantee that the GC system is contaminant free.

Supelco recommends the use of four types of gas purifiers. Table 1 describes these purifiers and the contaminants they remove.

Table 1. Purifiers and the contaminants they remove						
Name	Contaminants Removed	Size	Fitting	Cat. No.		
Supelcarb™ HC	C3 and higher hydrocarbons	120cc	1/8″	24448		
		120cc	1/4″	24449		
		750cc	1/4″	24564		
		750cc	1/2″	24565		
Molecular Sieve	H ₂ O	200cc	1/8″	20619		
5A Traps		200cc	1/4″	20618		
		750cc	1/4″	23991		
		750cc	1/2″	23992		
High Capacity	O ₂ , H ₂ O, CO ₂ & CO	32cc	1/8″	22396		
Heated Purifier**			1/4″	22398		
OMI™ -2	O ₂ , H ₂ O, CO ₂ , CO, NH ₃ ,	15cc	1/8″	23906		
	alkynes, halocarbons, halogens,					
	& Hydrogen halides					
** Can be used with He, N_2 , Ar/CH ₄ not compatible with air, O_2 or H_2 .						

Proper Sequence of Gas Purifier Installation

The proper sequence of purifier installation is important for peak performance. Remove hydrocarbons first, followed by moisture, and then oxygen. Hydrocarbons bind with moisture and oxygen removal sites in moisture and oxygen traps reducing their effectiveness. Likewise, water binds to oxygen removal sites in oxygen traps reducing

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FEATURED PRODUCTS

High Capacity Gas Purifier



OMI Indicating Purifier



Molecular Sieve 5A Water Vapor Traps



Therm-O-Ring Seals / Inlet Seals for Aailent/HP GC's



Micro-Flo 20 Flowmeter



Shortix Fused Silica Tubing Cutter



Gas Generation, Purification and Delivery

High Capacity Gas Purifier

A single, replaceable, High Capacity Gas Purifier tube can remove 14 liters of oxygen or 35 liters of water vapor (at STP). It removes oxygen and moisture from at least 60 cylinders of heavily contaminated gas. The High Capacity Gas Purifier can operate at flow rates up to 1100mL/ min., and is compatible with any common carrier gas except hydrogen.

110V 1/8" Fittings	23800-U
110V 1/4" Fittings	
230V 1/8" Fittings	
230V 1/4" Fittings	
Pressure Gauge Kit	
S For more information request T400205	

For more information, request T400205.

OMI Indicating Purifier

The OMI purifier provides point-of-use gas polishing and final visual assurance of gas quality before the gas enters the GC. The OMI purifier tube contains Nanochem™ resin developed to meet the demanding requirements of the semiconductor industry. As little as 1ppm of oxygen will change the indicating resin from brown to black. The OMI purifier tube, tube holder, and seal kit are needed for proper installation.

OMI-2 Purifier Tube	23906
OMI-2 Tube Holder	23921
Seal Kit for OMI Tube Holder	23917
S For more information, request T400205.	

Molecular Sieve 5A Water Vapor Traps

Install this inexpensive drying tube upstream of the High Capacity Purifier. The Molecular Sieve 5A Moisture Trap reduces water contamination under 0.5ppm. Installed before the OMI purifier, it reduces water in electrolytically generated hydrogen to less than 0.5ppm for a full year. With compressed house air for FID, the molecular sieve by itself will remove excessive moisture and heavy hydrocarbons.

200cc, 1/8" fittings	20619
200cc, 1/4" fittings	20618
750cc, 1/4" fittings	23991
750cc, 1/2" fittings	23992
S For more information, request T400205.	

Sample Preparation and Introduction

Therm-O-Ring[™] Seals

High pressure Therm-O-Ring inlet seals for Agilent[™] inlet liners provide superior GC performance at temperatures as high as 375°C. Supelco's proprietary formulation yields O-rings that do not stick to the injection port or fragment during removal. These rings are a superior replacement for Viton O-rings and are available exclusively from Supelco. Therm-O-Ring Seals, Pk. of 10 21003-U Therm-O-Ring Seals, Pk. of 25 21004-U **S** For more information, request T400003.

P000300

All literature mentioned in this issue can be obtained from the website, www.sigma-aldrich.com/TheReporter, by completing the Literature Request section on the reply card, or by calling our Technical Service Department.

Inlet Seals for Agilent GC's

Low cost, replacement inlet seals for Agilent GC's from Supelco reduce the need for cleaning and reuse. Supelco metal selection yields a better inlet seal. Seals are available in stainless steel and gold plated versions. Precise, computerized machining reduces dimensional variation that can occur with other seals.

Stainless Steel HP Inlet Seals, Pk. of 2	23316-U
Stainless Steel HP Inlet Seals, Pk. of 10	23317-U
Gold Plated HP Inlet Seals, Pk. of 2	23318-U
Gold Plated HP Inlet Seals, Pk. of 10	23319-U
E	

S For more information, request T400006.

Fittings and Accessories

Micro-Flo 20 Flowmeter

A more accurate and less time consuming method of measuring capillary flow, the Micro-Flo 20 electronic flow meter is designed to provide continuous, accurate linear velocity and volumetric flow of 20ml or less for helium and hydrogen. It is particularly useful for setting flows for methaneretaining columns or when using detectors that do not respond to methane. It also eliminates errors introduced when using nominal column lengths to calculate linear velocity. Laminar Micro-Flo 20 Flowmeter 23144 **S** For more information, request T496002.

Shortix[™] Fused Silica Tubing Cutter

A revolution in capillary column cutting, the Shortix Fused Silica Tubing Cutter allows practically anyone, regardless of skill level, to complete near perfect capillary column cuts every time. The Shortix fused silica tubing cutter uses a diamond cutting edge that rotates around the column etching the entire surface of the fused silica tubing. This yields a consistently clean edge that improves your ability to make leak free connections. Shortix Fused Silica Tubing Cutter 21386-U

S For more information, request T498335.

Columns

Capillary Columns for Agilent 6850

Supelco makes it easy to purchase off-the-shelf capillary GC columns for the Agilent 6850! By referencing order code PRO100060, you can purchase any stock or custom Supelco capillary column wound on an authentic Agilent Technologies 6850 cage. To order, simply provide order code PRO100060 plus the stock capillary item number or custom column information. Supelco will coil the column onto a 6850 cage and ship the column within 24 hours.

S For more information, request T400051.

APPLICATIONS

Automated Air Sampling using ASSET-32 Tubes

A charcoal solid phase extraction (SPE) tube, ASSET-32 allows users to simplify sample prep for air sorbent tubes. The ASSET-32 configuration allows elution directly to an autosampler vial that eliminates the need to remove the charcoal from the tube. This poster session was presented at the June AIHCE conference in Salt Lake City, UT.

S For more information, request T400126.

Air Monitoring Applications Using XAD-2[®] And Silica Gel Adsorbents In A Timesaving Air Sampling Solid Phase Extraction Tube (ASSETTM)

Following NIOSH 5515 the ASSET-43 was evaluated for naphthalene and benzo(a)pyrene by GC/FID and comparisons made with a standard glass tube containing the same amount of XAD-2. NIOSH 2005 was evaluated for nitrobenzene with the same comparisons made with a glass tube. Background levels, a recovery study over four concentration levels, and a storage stability study was performed for both tubes. Performance criteria were targeted at $\leq 10\%$ difference to the glass tube equivalent. This poster session was presented at the June AIHCE conference in Salt Lake City, UT.

S For more information, request T400127.

SEMINARS

A Systematic Approach for Selecting the Appropriate SPME Fiber

This presentation, presented at the ExTech Conference, describes the difference between adsorbent and absorbent type fibers. It also discusses the various types of SPME fibers commercially available. This updated presentation includes notes from the presentation and is available in printed form or in Adobe Acrobat[®] format.

S For further information, request T400156.

GC PERFORMANCE TIP

Change Gas Cylinders Early to Extend Purifier and Column Life

The tank pressure at which you decide to change your gas cylinders may affect the quality of your carrier gas. The lower the pressure, the greater the risk of causing problems with your chromatography system and your column due to higher levels of tank contaminants.

When the pressure in your gas cylinder drops below 500 psig, you risk drawing higher levels of contaminants from liquids that may be present in the bottom of the cylinder. You increase the chance of adding oxygen, moisture, and other contaminants to your carrier gas. These contaminants will shorten the lifetime of your purifiers and column.

NEW LITERATURE

Supelco Specialty Packaging Services

Repackaging and reference material management are critical links in the drug development supply chain. You can make your chain stronger by linking with Supelco's Specialty Packaging Services. For 100 or 100,000 units, Supelco lets you do it faster, make it better, and do it for less money. This brochure describes Supelco's Specialty Packaging Services.

S For more information, request T400119.

MS-No Vent

A new time saving accessory for GC/MS systems, the MS-NoVent allows capillary users to begin using the mass spec within minutes after changing columns. The MS-NoVent supplies the mass spec with carrier gas during column change, eliminating system pump down. It consists of a pressure switching valve, fused silica restrictor, and an external control module. Installation on Agilent/HP, Varian, Shimadzu, and other systems is typically less than 30 minutes. A new Product Information Sheet is available describing this valuable device for GC/MS users.

S For more information, request 400005.

Supelco Petroleum Guide Updated

This 48-page guide contains information about Supelco products and technology for separating hydrocarbons by chromatographic methods. The updated guide illustrates nearly one hundred different hydrocarbon separations including PONA, PIANO, and SIMDIS applications. Multiple capillary and HPLC products are included to help with your analytical hydrocarbon needs. The guide was recently updated to reflect current products and applications available at Supelco.

S For more information, request T100858.





Ron Shawley -GC Product Specialist

chromatography will be the result. Supelco recommends that you change your gas cylinders when they drop to a pressure between 500 and 300psig. Avoid the risk of using impure carrier gas and causing more costly problems.

Additionally, two-stage pressure regulators do not operate

effectively at pressures less than 300psig. Below 300psig, your two-stage regulator begins to function more like a single-

stage regulator, resulting in variable pressure. Without constant pressure, retention times will vary and irreproducible **Bulletin 918** (T197918) includes information needed to select suitable purifiers for carrier gas, and for air and hydrogen used as fuel gases.

Bulletin 898 (T196898) This bulletin, in combination with the instruction manual for your chromatograph, will help you to use your gas management resources wisely. We recommend you read this bulletin before installing your GC.

CASE

STUDY

Trademarks and Registered

Adobe Acrobat - Adobe Systems,

Agilent - Agilent Technologies, Inc.

Supelcarb, Supeltex, Supelcowax,

domnick hunter, Nitrox - domnick

HP - Hewlett-Packard Corporation

Shimadzu - Shimadzu Corporation Shortix - SGT-Middleburg, B.V. Co.

Teflon - E.I. du Pont de Nemours &

Viton - DuPont Dow Elastomers

exclusively to Supelco. US patent

#5,691,206; European patent

MS-NoVent - Scientific Glass

Nanochem - Matheson Gas

Perkin-Elmer - Perkin-Elmer

Varian - Varian Associates

SPME - Technology licensed

Thermogreen, Therm-O-Ring,

Carbowax - Union Carbide

CapSeal Bullet, OMI, SPB,

Trademarks:

Sigma Aldrich

hunter Limited

Engineering

Corporation

Products

Co., Inc.

Patents:

#523092.

Inc

Proper Column Installation...

(continued from page 1)

their effectiveness. Figure A illustrates the proper sequence of installation for carrier gas purifiers.

Purifiers improve the quality of even the highest purity carrier gas. As a final safeguard, Supelco recommends installing an OMI (Oxygen-Moisture Indicator) purifier tube just before the carrier gas line entering the GC. This protects against contaminants that may have entered through fittings in the gas lines. The OMI tube also gives a visible indication of contaminants. If it changes color, you know the GC system has leaks or that the purifiers have expired and need replaced.

The Unstable Baseline

Background

Joe Narrowpeaks joined a GC food group after many years of experience in hydrocarbon analysis. In his new job, he analyzes fish oils as fatty acid methyl esters (FAMEs). Joe uses Carbowax[®] PEG capillary columns with temperature programming to provide carbon number and degree of unsaturation separations of FAMEs.

The Problem

Joe soon notices an odd shaped "hump" eluting in many of his analyses. At times, unidentified peaks appear as part of the hump. Joe sees this hump even when making a blank injection. Figure B illustrates the chromatography. One of the other chemists in the lab comments that the hump and unknown peaks are probably "garbage" from the column.



Joe notes that the hump and unknown peaks are increasing steadily in size. Once the interference becomes large enough, Joe is no longer able to complete his analysis. He notes that the PEG columns have a significantly shorter lifetime in comparison to methylsilicone columns he used in his previous job.

Conclusion

You need carrier gas purifiers regardless of the gas purity used. Contamination is always present in the GC system. Sources include the gas cylinder, the cylinder changing process, fittings and regulators. Carrier gas purifiers are the only sure way to guarantee the lowest level of carrier gas contamination. Install purifiers in the right sequence for proper purification. Installation must include a hydrocarbon trap, followed by a high capacity moisture and oxygen purifier, and finally an OMI indicating purifying tube. Maximum GC column life is possible only if you use the right carrier gas purification products.

S For more information, request T197918 and T196898.

Missing Carrier Gas Purifiers

Remembering that polyethylene glycol (PEG) phases are highly susceptible to damage from moisture and oxygen, Joe decides to evaluate his GC system. He is using Agilent 6890's with split/splitless injectors and auto-injection systems. The Helium carrier gas supply is 99.999%; the same grade Joe used at his old job. As he continues his review, Joe cannot locate carrier gas purifiers anywhere in the system. Knowing that carrier gas contaminants can damage columns, Joe decides to install gas purifiers.

Joe orders the recommended carrier gas purification supplies from Supelco. He installs hydrocarbon, moisture, and oxygen traps down stream of the manifold system. Purifier components include a Supelpure hydrocarbon trap, a molecular sieve moisture trap, and a high capacity heated purifier. Joe equips each GC with an OMI indicating purifier. He installs the OMI tubes in the carrier gas lines within eighteen inches of the final GC connection.

Figure C. Baseline after purification



Results are Almost Immediate

Within several runs of adding carrier gas purifiers, Joe notices that the hump and interfering peaks are much reduced. The next day, Joe gets a flat baseline when making a blank injection. Figure C illustrates Joe's new chromatography.

Over the next few weeks, Joe notes that the PEG columns are exhibiting more stable performance. Joe concludes that adding carrier gas purifiers improved his system performance.

S For more information, request T400205.



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